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FOREST PRODUCTS LABORATORY

In cooperation with the University of Wisconsin

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PULPING EASTERN HEMLOCK BY THE SULPHITE PROCESS

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Introduction

Although the consumption of eastern hemlock by sulphite mills in the Lake States region has increased during the past few years, mainly as a result of the scarcity and the high price of domestic spruce, no literature bearing on the question of optimum cooking conditions for the species has been made available. Accordingly experimental studies in the pulping of eastern hemlock by the sulphite process were undertaken at the U. S. Forest Products Laboratory. The penetration period was selected as the first subject for investigation because it is now recognized as a separate and extremely important portion of the total pulping cycle. The immediate objective of the studies was to determine if possible the optimum conditions for penetration of the chips as shown by the yield of screened pulp, the amount of screenings, and the strength qualities and bleach requirements of the pulps produced. This paper, the first of the series presenting the information derived, sets forth the results obtained in a group of semi-commercial-scale pulping experiments in which either the time or the temperature of the penetration period was varied, other factors being held constant.

Previous Work

During the past few years the Laboratory has made many pulping tests on eastern hemlock and similar species, but the work has been of an exploratory nature and as a result no particular effort was made to arrive at optimum pulping

conditions. On the other hand, the results so obtained have been valuable as a guide in this study.

Characteristics of Wood

The species of the wood employed in the experiments was identified as Tsuga canadensis. The pulp wood used for the tests consisted of tops cut from trees grown in northeastern Wisconsin. In order to obtain an idea of the uniformity of the wood as well as to record certain physical properties, specific gravity tests were made on disks cut from a number of the logs. The specific gravity values are expressed as oven-dry weight of green volume. The samples, which were taken at random, represented approximately 30 per cent of the total logs used. The age of each specimen was determined by counting the annual rings. The average diameter of the disk inside the bark was also determined. A value for average growth rate was obtained by dividing the number of annual rings by the radius of the bolt. All these values are given in Table 1.

Specific Gravity Varies Widely

The tabulation makes evident the fact that the specific gravity of the wood, which influences considerably the penetration of the chips by the acid cooking liquor, varied widely. For spruce and balsam fir it has been shown[1] that there is a relationship between the average growth rate and the specific gravity of the bolts. This relationship did not hold for the eastern hemlock examined.

In order to obtain as much uniformity as possible in the material for the various cooks, the chips forming the total supply were thoroughly mixed before any of the experimental cooks were made.

Temperature and Time Schedules

The temperature and time schedule, as pointed out in the pulping of spruce[2] and of hardwoods,[3] has been shown to be

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[1] Pulp and Paper Mag. of Canada, International Number, 139-149 (Feb., 1923).
[2] Paper Trade J. 82, No. 9, 62-64 (1926).
[3] Paper Trade J. 86, No. 17, 59-62 (1928).

Table 1.--Physical properties of eastern hemlock pulp wood

Disk No.	Specific gravity (oven-dry volume)	Weight per cubic foot (oven-dry)	Age (annual rings)	Average diameter (barked and oven-dry)	Annual rings per inch
		Pounds	Years	Inches	
1	0.386	24.1	100	8.37	35.40
2	.448	28.0	168	8.00	56.00
3	.497	31.1	130	8.56	39.70
4	.419	26.2	46	5.87	15.70
5	.429	26.8	188	8.87	54.80
6	.416	26.0	159	7.31	43.30
7	.452	28.2	157	7.56	41.60
8	.406	25.4	98	6.87	28.60
9	.479	30.0	212	4.93	86.00
10	.573	35.8	150	5.00	60.00
11	.424	26.5	66	5.32	25.80
12	.491	30.7	110	4.37	50.10
13	.366	22.9	49	8.50	15.06
14	.473	29.5	181	7.00	51.80
15	.406	25.4	105	8.37	32.90
16	.415	25.9	135	7.12	37.90
17	.401	25.0	165	7.50	44.00
Average	0.440	27.5	130	8.54	41.00

one of the major factors in the pulping of any species by the sulphite process. In this series of experiments both the time and the temperature of the penetration period were varied in turn, one of the two factors always remaining constant.

The pulping schedules followed from the end of the penetration period to the maximum temperature, 148 degrees C., were all of seven hours duration and of the same type, namely, the so-called "straight-line rise." The temperature at the beginning of the pulping period varied, depending upon the highest temperature of the penetration period. Three such high temperatures were used, 110 degrees, 115 degrees, and 120 degrees C. The time required to reach these temperatures was varied as follows: 1.5, 2.0, 2.5, 3.5, and 5 hours to 110 degrees C., 1.5, 2.0, and 2.5 hours to 115 degrees C., and the same to 120 degrees C.

The effects of the variations in procedure just described were recorded by determining the yield of screened pulp and screenings, the bleachability, and the strength development of the pulps produced.

Experimental Equipment and Procedure

The experimental cooks were all made in the Laboratory's semi-commercial sulphite digester,[4] which has a capacity of about 85 pounds of oven-dry chips.

The cooking acid used was adjusted so as to approximate 5.50 per cent total sulphur dioxide, of which about 1.30 per cent was present as combined sulphur dioxide.

In order to counteract the excessive condensation of steam caused by the abnormal radiation from the small-sized equipment, both direct and indirect steam heating were used for the digester. The steam for direct heating was so regulated in amount that the dilution effect of the condensed steam was similar to that obtained in commercial "quick-cook" practice. This variable is under control by observation and test of the relief liquor.

Temperature and Pressure Schedule

The rate of temperature increase and the pressure schedule for each cook are shown in Figures 1, 2, 3 and 4. A

[4] A complete description of the equipment may be found in U.S. Department of Agriculture Bulletin No.1485, 11-15 (1927).

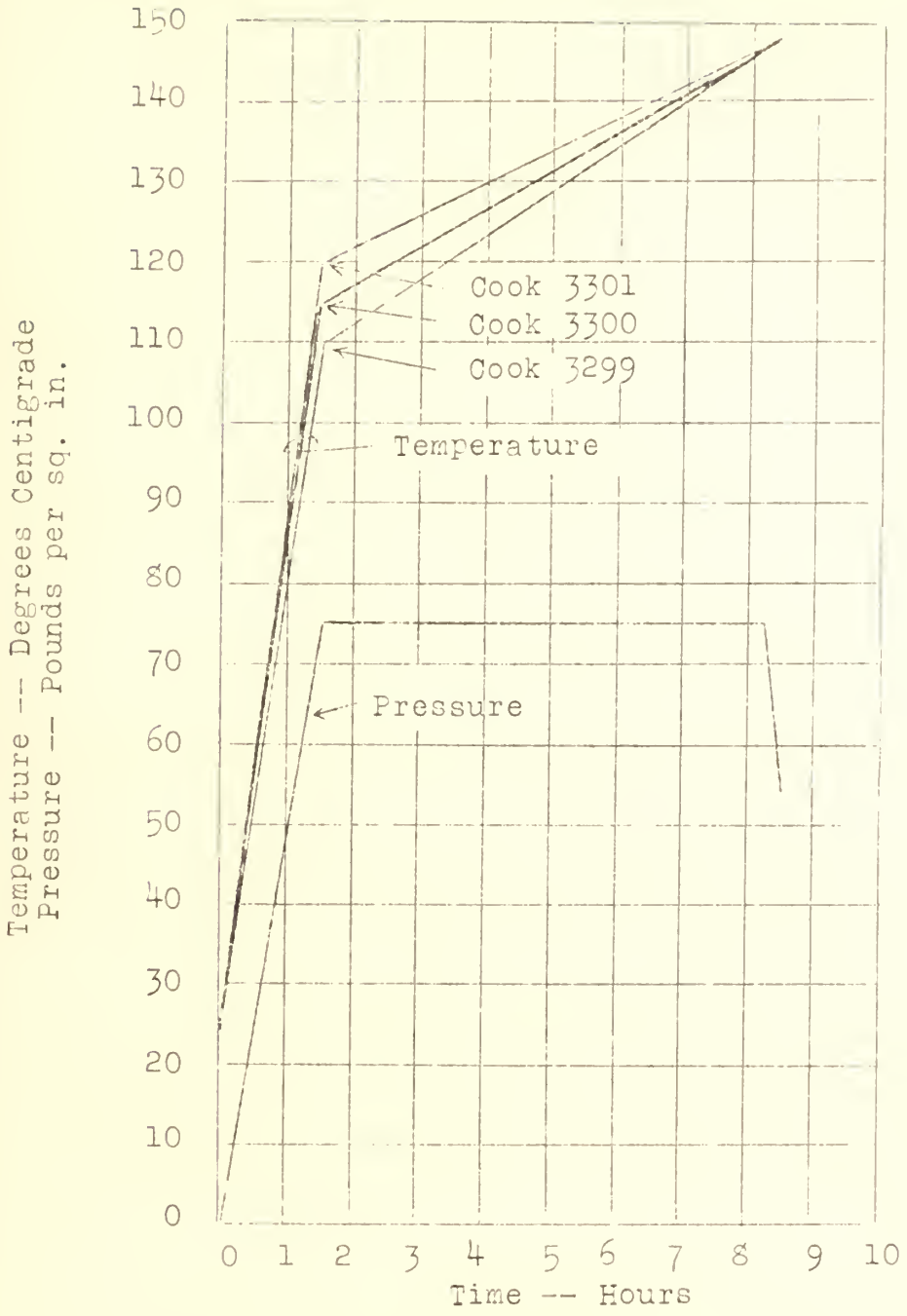

Fig. 1.--Temperature-pressure chart for 1.5 hours penetration period.

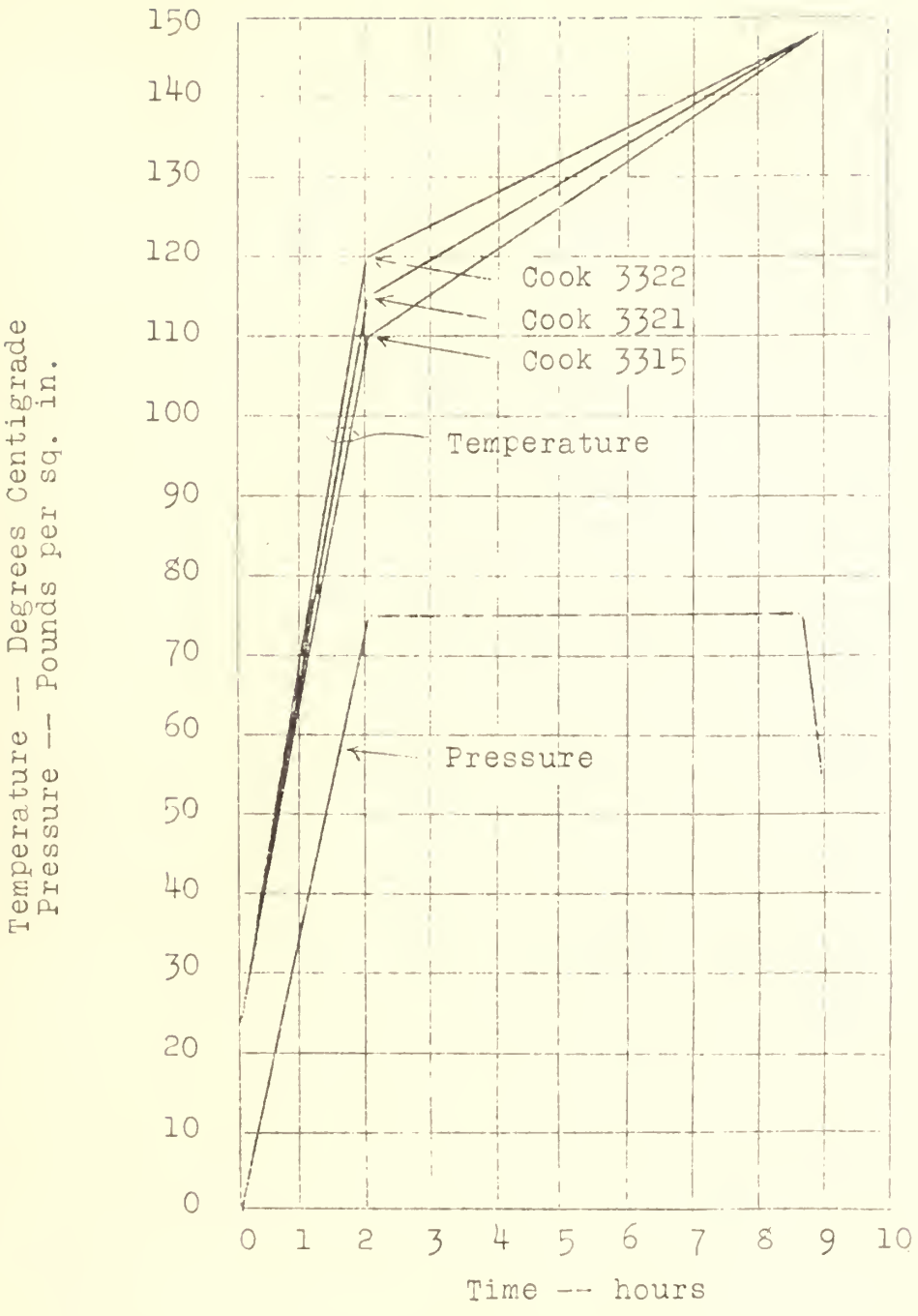

Fig. 2.--Temperature-pressure chart for 2.0 hours penetration period.

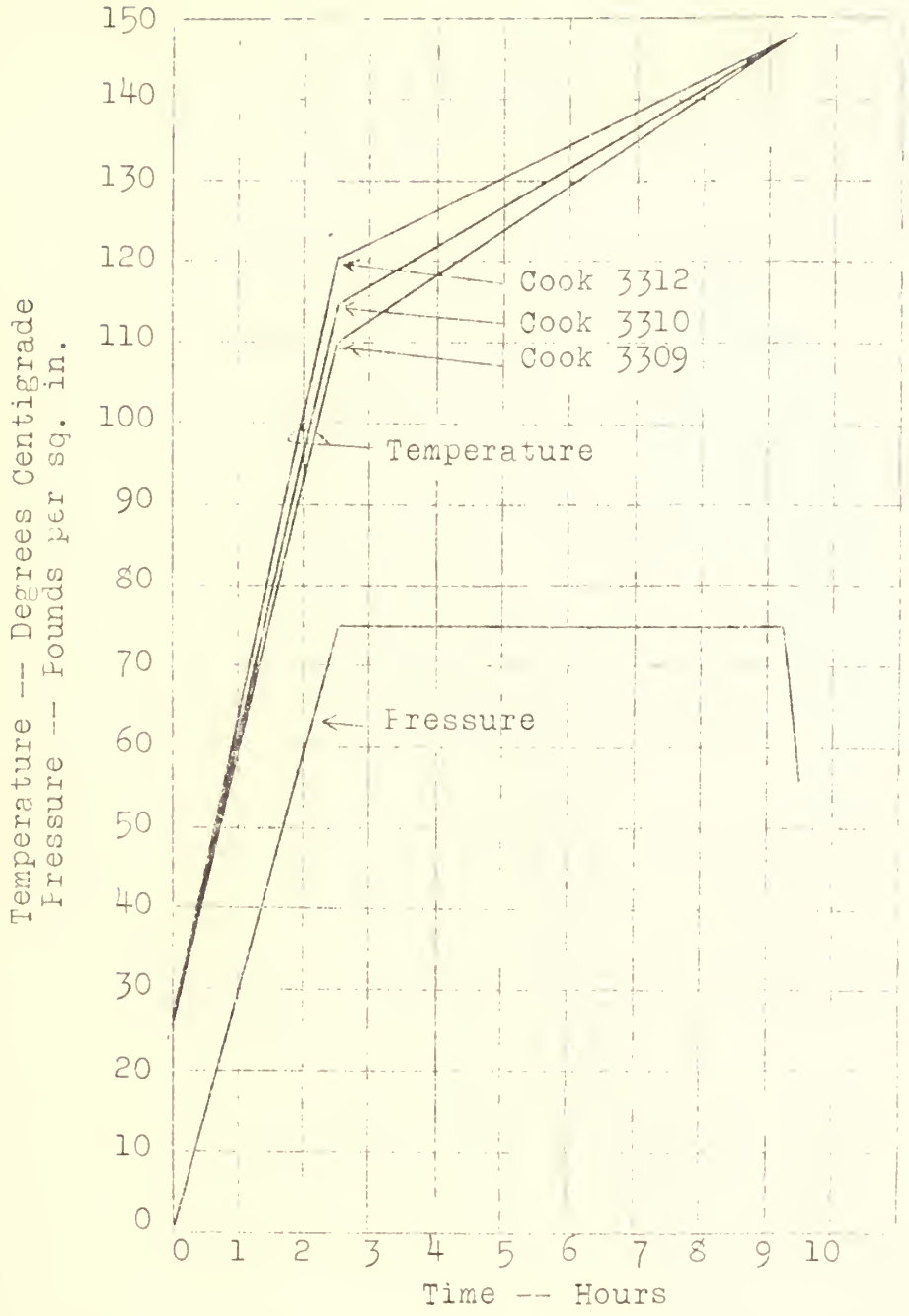

Fig. 3.--Temperature-pressure chart for 2.5 hours penetration period.

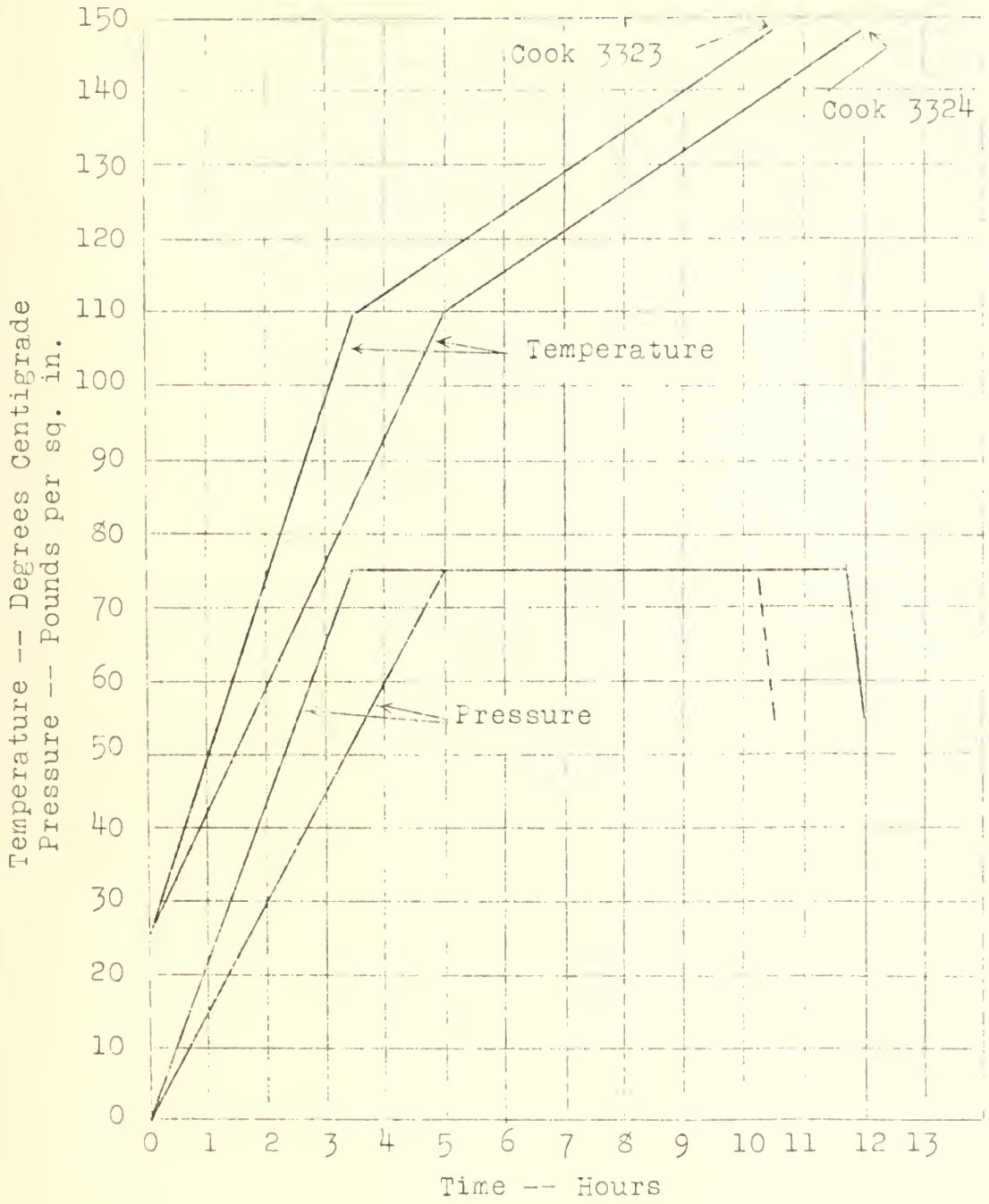

Fig. 4.--Temperature-pressure chart for 3.5 and 5 hours penetration periods.

maximum pressure of 75 pounds was used throughout, followed by a uniform relief period at the completion of the cook.

Yield determinations were made by screening the pulp over a diaphragm screen having 0.012-inch slots. Samples for moisture determination were taken at regular intervals as the pulp was run over the experimental wet machine. Knowing the total weight of wet pulp and the moisture content, the oven-dry weight of pulp was calculated. Screenings were oven-dried and weighed.

The bleach requirement of the various pulps were determined by treating a sample equivalent to 25 grams (oven-dry weight) of pulp, in a water suspension at a constant consistence of 1.5 per cent, with varying ratios of bleach powder to pulp. Temperature was maintained constant at 35 degrees C. and the stirring was continuous until the chemical was exhausted. The pulp was then thoroughly washed, made up into hand sheets, and dried. After careful pressing of the dry sheets, color measurements were made on the samples by the use of the Hess-Ives tint photometer. Samples of bleached Burgess sulphite pulp were used as a standard for comparison. The bleach requirement was taken as the amount of bleach powder needed to bleach the sample of pulp to the same degree of whiteness as that of the standard.

The ability of the pulps to develop fiber strength was determined by the ball-mill method as standardized at this Laboratory. Test sheets were prepared from the unbleached pulps after 20, 40, 60, and 80 minutes milling. Bursting-strength, folding-endurance, and tearing-strength tests were made for each milling period and each cook.

Tabulation of Results

The pulping conditions, pulp yields, and bleach requirements are presented in detail in Table 2.

The data on the development of fiber strength by ball-milling the unbleached pulps are presented in detail in Table 3.

Discussion of Results

As previously stated, the main emphasis in the experimental work performed thus far has been placed on the

Table 2.--Cooking conditions employed, and yields and bleach requirements of hemlock sulphite pulps[1]

Cook No.	Temperature and pressure curve with time	Acid composition[2]			Total time of penetration period	Max. temperature at end of penetration period	Total[3] cooking cycle including penetration period	Yield of oven-dry pulp, based on oven-dry weight of wood			Bleach requirement, based on 35 per cent bleach powder
		Total SO_2	Free SO_2	Combined SO_2				Total pulp	Screened pulp	Screenings	
	Reference figure	Per cent	Per cent	Per cent	Hours	°C.	Hours	Per cent	Per cent	Per cent	Per cent
3299-I	1	5.55	4.27	1.28	1.5	110	8.5	44.0	40.6	3.40	35+
3300-I	1	5.50	4.18	1.32	1.5	115	8.5	44.25	40.6	3.65	28
3301-I	1	5.50	4.18	1.32	1.5	120	8.5	44.15	40.9	3.25	28
3315-I	2	5.50	4.20	1.30	2.0	110	9.0	48.25	45.85	2.40	34+
3321-I	2	5.50	4.18	1.32	2.0	115	9.0	45.95	43.6	2.35	30
3322-I	2	5.60	4.30	1.30	2.0	120	9.0	45.50	43.1	2.40	28
3309-I	3	5.50	4.20	1.30	2.5	110	9.5	46.80	44.2	2.60	34+
3310-I	3	5.55	4.29	1.26	2.5	115	9.5	47.80	45.6	2.20	30
3312-I	3	5.50	4.19	1.31	2.5	120	9.5	45.60	43.1	2.50	30
3323-I	4	5.50	4.25	1.30	3.5	110	10.5	45.75	43.7	2.45	32+
3324-I	4	5.40	4.08	1.32	5.0	110	12.0	44.15	42.7	1.45	26

[1] Maximum cooking temperature held constant at 148° C.
Maximum cooking pressure held constant at 75 pounds gauge.

[2] Acid composition held as nearly constant as possible.

[3] The pulping period following the impregnation period was held constant at seven hours duration.

Table 3.--Development by ball milling of the fiber strength of unbleached hemlock sulphite pulp

Cook No.	Strength-test results: Bursting-strength factor in points per pound per ream of 24" x 36", 500 sheets, when refined in the ball mill for				Folding endurance in double folds when refined in the ball mill for				Tearing-strength factor in grams per pound per ream when refined in the ball mill for			
	20 min.	40 min.	60 min.	80 min.	20 min.	40 min.	60 min.	80 min.	20 min.	40 min.	60 min.	80 min.
3299-I	---	0.75	0.96	0.95	---	227	347	483	---	2.39	2.30	1.96
3300-I	0.49	0.72	0.82	0.85	37	144	279	384	2.30	2.63	2.36	2.03
3301-I	0.50	0.69	0.82	0.87	58	98	351	461	2.24	2.32	2.57	2.22
3315-I	0.65	0.87	1.02	1.02	43	207	197	334	2.32	2.31	2.17	1.99
3321-I	0.74	0.90	0.97	0.90	140	214	318	350	2.59	2.67	2.39	2.20
3322-I	0.57	0.80	0.94	0.78	92	180	538	425	2.71	2.61	2.49	2.05
3309-I	0.68	0.90	0.97	0.97	104	294	501	382	2.85	2.68	2.39	2.21
3310-I	0.69	0.88	1.00	0.88	39	223	402	505	2.12	2.23	2.11	1.84
3312-I	0.67	0.74	0.96	0.92	58	250	621	517	2.35	2.61	2.22	2.30
3323-I	0.70	0.91	1.01	0.96	105	233	448	420	2.51	2.75	2.42	2.41
3324-I	0.65	0.84	0.96	0.90	80	295	251	472	2.46	2.71	2.25	2.20

The maximum values are underscored throughout the table. Where the 80-minute value is underscored, increased duration of milling is necessary to determine whether this value is in reality the maximum.

investigation of the penetration period by varying its time and the temperature employed; the effects of such variation were gauged by the yield of screened pulp, the amount of screenings, and the strength qualities and bleach requirement of the pulps produced.

Effect Upon Yields

Increasing the temperature at 1.5 hours time had no effect on the total yield of pulp and the percentage of screenings. The total yield of pulp for each of the three cooks was low and the screenings were comparatively high. On the other hand, increasing the temperature at 2.0 and 2.5 hours had a decided effect on the total yield of pulp. The yields of total pulp and of screened pulp decreased with no change in the percentage of screenings.

Maintaining the temperature constant at 110 degrees C. and increasing the time of the penetration period resulted in an increase in the total yield of pulp and a decrease in screenings. For the 5-hour period the total yield of pulp was slightly lower than had been expected. Even though the total yield was low the screened pulp was 2 per cent higher and the screenings were 2 per cent lower than the corresponding yields obtained for the experiment in which 1.5 hours was used for penetration.

Increasing the time at 115 degrees C. and 120 degrees C., respectively, resulted in an increase of total yield of pulp and a decrease in screenings. Pulping experiments having longer periods of penetration than 2.5 hours were not made at the higher temperatures.

The pulps of highest yields and lowest percentage of screenings were obtained from the experiments in which a 2-hour or longer period was used for penetration. The maximum temperature of penetration seems not to affect the yield properties very markedly, except when short penetration periods are used. A temperature of 115 degrees C. may be used as the maximum temperature with good results.

Effect Upon Bleachability

An increase in the maximum temperature for any penetration period of constant length results in a lowering of the bleach requirement of the pulps.

Holding the temperature constant at 110 degrees C. and increasing the time of penetration from 1.5 to 2.5 hours had no effect on the bleach requirement of the pulps. A further increase in time of penetration from 2.5 to 5 hours lowered the bleach requirement from 34+ per cent to 26 per cent.

A slight increase in the bleach requirement resulted as the temperature was held constant at 115 degrees C. and 120 degrees C., and the time increased. For all practical purposes this increase in bleach requirement may be neglected, since it was only 2 per cent.

The optimum conditions in regard to bleach requirement of the pulps are obtained by utilizing the higher temperatures (115 degrees C. and 120 degrees C.) for periods of 1.5, 2.0, and 2.5 hours, or by extending the time at the lower temperature (110 degrees C.) to at least 5 hours.

Effect Upon Bursting Strength

The bursting-strength factor of pulps from experiments in which a temperature in excess of 110 degrees C. is reached in 1-1/2 hours time are somewhat low. Increasing the temperature at longer periods apparently has no effect on the bursting properties of the pulps. The maximums are reached after 60 minutes refining in the ball mill.

With an increase in time, and the temperature constant at 110 degrees C., no increase in the maximum bursting-strength factor was noted. On the other hand, an increase in bursting properties resulted upon increasing the time at temperatures of 115 degrees C. and 120 degrees C.

The conditions that yield pulps with maximum bursting properties indicate that 2 to 2-1/2 hours time is essential for the increase to a temperature of 110 degrees C. to 120 degrees C.

Effect on Folding Properties and Tearing Strength

There are no general trends in the folding-endurance or tearing-strength factors of the pulps with increased temperature or time.

Summary

The results obtained in this series of pulping experimenta may be summarized as follows:

The yields of pulp decrease with an increase in temperature during the penetration period, the amount of screenings remaining practically constant.

With an increase in the time of the penetration period the yields of pulp increase for temperatures of 115 degrees C. and 120 degrees C., but fail to do so for 110 degrees C.

A decrease in screenings is obtained by increasing the time of the penetration period.

A definite reduction in the bleach requirement of the pulps is obtained with an increase in temperature of the penetration period.

The bursting strength factor decreases as the temperature of the penetration period is increased from 110 degrees C. to 115 degrees C. and 120 degrees C., at 1-1/2 hours time.

As the time of penetration is increased for 115 degrees C. and 120 degrees C., respectively, the bursting strength factor increases.